



# Investigation of Residue and Coating Stoichiometry on 120-mm Combustible Cartridge Cases

by Kestusis G. Chesonis, Pauline M. Smith, and William S. Lum

ARL-TR-2337

September 2000

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**ARL-TR-2337****September 2000**

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## **Investigation of Residue and Coating Stoichiometry on 120-mm Combustible Cartridge Cases**

**Kestutis G. Chesonis, Pauline M. Smith, and William S. Lum**  
Weapons and Materials Research Directorate, ARL

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## Abstract

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An investigation was conducted to determine the cause of coating residue found in the test gun chambers during qualification firing of 120-mm combustible cartridge case (CCC) ammunition for the M1A1/A2 main battle tank. The CCC is coated with a clear epoxy-polyamide topcoat and a highly pigmented aluminum epoxy basecoat. A laboratory analysis was performed on actual residues; parts of the CCC retain samples of actual coatings and laboratory control samples. Since there are six different resins used in this two-component coating system, production errors and deviations from correct mixing prior to application could have caused the residue. Any variation from the specified stoichiometry could also result in films with lower cross-link densities and lower glass transition temperatures ( $T_g$ ). Rounds fired at 120 °C have more residues than identical rounds fired at ambient temperature; additional weeks of cure lower the amounts of residue. A differential screening calorimeter (DSC) analysis of the various samples revealed that a suspect lot of ammunition with higher levels of residue had lower  $T_g$  values. Fourier-transform infrared (FTIR) was effective in monitoring the degree of cure of the epoxy coating system. Analyzing the residue from actual firings showed similar features. Koenig hardness values showed variations caused by dry film thickness, but also showed insignificant changes caused by transformations in the stoichiometry of the clear topcoat from 52.5 to 45 phr (pounds per hundred of resin).

## **Acknowledgments**

The authors would like to thank Jae Shim and the U.S. Army Armament Research, Development, and Engineering Center (ARDEC), Picatinny, NJ, for sponsoring the study. We also thank Philip Patterson, Gume Rodriquez, Jeffrey Duncan, and Alan Teets of the U.S. Army Research Laboratory (ARL) for their assistance. A special thanks to Dr. Alex Hsieh of ARL for his hard work, technical comments, and guidance on this project.

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# 1. Introduction

The 120-mm family of tank ammunition used by the U.S. Army's main battle tank, the Abrams M1A1/2, is the most advanced and lethal in the world. The two main types of ammunition are the kinetic energy (KE) and the high explosive anti-tank (HEAT); both have the propellants in an aluminum-coated combustible cartridge case, and only a small metal base case is discharged after firing.

A major deficiency of the coating that existed in 1990 was its tendency to delaminate in sections when subjected to stress during uploading and downloading (Koehler 1990). He pointed out that premature ignition from these unprotected areas was possibly caused by their susceptibility to any burning residue left in the gun chamber. Another area of concern was that moisture penetrated the combustible cartridge in areas that lacked protective coatings. For these reasons, Picatinny Arsenal, NJ, requested that The Coatings Team evaluate this problem and recommend solutions.

The final product looks similar to the type of metallic finish on many automobiles. The performance specification requires the coating system to be tough yet flexible, and able to endure repeated uploading and downloading during training and actual operations. The basecoat provides resistance to moisture and ignition from any possible residue left from previous firings, while the topcoat provides toughness and film integrity. To avoid any stress-induced delamination, coatings were formulated to balance the coefficient of thermal expansion and the elastic modulus between the basecoat and topcoat. Since these coatings are thermosets in nature, the stoichiometric ratio of the components and the degree of cure play an important role in determining the physical and mechanical properties of the coatings on the finished rounds.

These coatings are applied by high-volume, low-pressure spray guns to the combustible cartridge cases (CCC), consisting of nitrocellulose, wood pulp, and polyurethane resin. The average film thickness is controlled to 30–35  $\mu\text{m}$  for the aluminum basecoat and 25–30  $\mu\text{m}$  for

the clear topcoat. Both the topcoat and basecoat are force dried at 160–180 °F for 20–30 min to ensure production and handling ruggedness.

The routine field testing conducted at the U.S. Army Test Center (ATC), Aberdeen Proving Ground (APG), MD, and at Yuma Proving Ground (YPG), AZ, involves firing coated 120-mm combustible cartridge cases conditioned at 120 °F, ambient, and subambient temperatures.

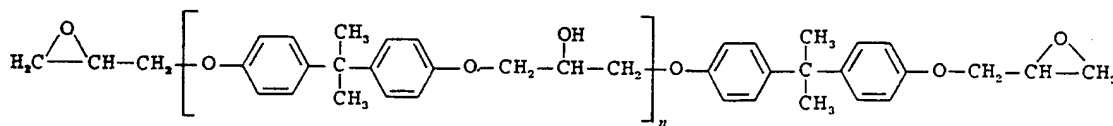
Field observations revealed that the coating residues left behind in the chamber after ballistic firings were significantly higher for the rounds that were conditioned at 120 °F. Excess residues were also evident when rounds were fired only seven days after a coatings application (not the usual time of 3–4 weeks). However, retesting a few weeks later gave acceptable results with minimal residue in all cases. A preliminary examination showed that the majority of these residues appeared to be the clear topcoat film with minor amounts of aluminum from the basecoat. Since the cross linkage of this epoxy system is continuous at ambient temperature, the industry-wide practice of evaluating film after a seven-day cure time is insufficient when the coating is subjected to high temperature and pressure in the gun chamber.

To establish feasible characterization techniques for the quality control of the coated cartridge cases, differential scanning calorimetry (DSC) and Fourier-transform infrared (FTIR) spectrometer characterization techniques were employed to determine the state of cure and cure chemistry of the clear topcoat. The focus was on identifying any differences in the coated specimens obtained from coating residues, production runs, production retains, and laboratory-prepared materials. The goal of this project was to identify and verify the causes of the firing residue and to provide a quick and reliable procedure for better quality control of the production rounds.

## 2. Formulation

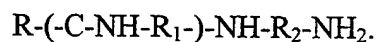
The topcoat epoxy is formulated at 52.5 parts by weight of the polyamide per 100 parts by weight of epoxy resin. This polyamide content is within the manufacturer's recommended ratio, 45:55, for high performance coating application. The resin composition is shown in Table 1. The structural formula for the epoxy resin is

### A. Bisphenol A-epichlorohydrin (DGEBA) resins



<i>n</i>	Trade names
0 (DGEBA)	Ciba-Geigy Araldite AY105, 6004, Dow DER 332, Shell Epon 828
2-3	Ciba-Geigy Araldite 6071, Dow DER 661, Shell Epon 1001
5-6	Ciba-Geigy Araldite 6084, Dow DER 664, Shell Epon 1004

while for the polyamide resin, it is



**Table 1. Resin Composition**

Resin	Total Solids	Viscosity	WPE (Epoxide equivalent weight) 100%	Amine Value
Epoxy resin	75%	Z <sub>2</sub> -Z <sub>7</sub>	450-550	—
Polyamide resin	70%	0.3-0.8 poise at 40 °C	—	161-173

## 3. Experimental

**3.1 Materials.** The U.S. Army Research Laboratory (ARL) Coatings Team received the following samples for evaluation:

- (1) One control cap and two from the lots with excessive residue, labeled cap No. 1 and cap No. 2. These caps attach the projectiles to the combustible cartridge cases.
- (2) Wet retains from the topcoat vendor, identified as component A and component B. Basecoat samples were not available.
- (3) Film thickness strips from the Iowa Army Ammunition Plant of various production lots, topcoat, basecoat, and a combination of both coats.
- (4) Two residues from an actual firing at Yuma Proving Grounds labeled as Yuma No. 7 and Yuma No. 8.

A sample of the polyamide-epoxy topcoat was prepared in the laboratory as a control. The formulation is given in Table 2. Films were allowed to cure at ambient temperature and were primarily used for establishing a baseline and for comparative purposes.

**Table 2. Topcoat Formulation**

Component A	
Ingredients	Ingredients (wt %)
Epoxy resin	57.9
Flow/leveling resin	0.8
Solvent A	19.7
Solvent B	5.6
Solvent C	16.0
Component B	
Ingredients	Ingredients (wt %)
Polyamide	38.8
Solvent A	14.5
Solvent B	43.1
Curing agent	3.6

Note: Mix ratio is 1:1 of part A to part B by volume.



**3.2 Thermal Analysis.** The glass transition temperature ( $T_g$ ) is one of the main characteristic temperatures of an amorphous solid. When an amorphous polymer is cooled or heated through the glass transition region, it undergoes structural relaxation.  $T_g$  can be defined as the temperature in amorphous polymers where a second-order phase transition is detected as a distinct increase in the rate of change of the polymer with temperature (TA Instruments 1997). All experiments were performed on a TA Instrument DSC 2920. DSC is an analytical method that measures the change in heat flow between the sample and an inert reference as a function of time and temperature (TA Instruments 1995). The selected sample and an inert reference are subjected to controlled conditions of time, temperature, atmosphere, and pressure. Both are placed in hermetic pans that sit on raised platforms on a constantan disk. Heat is transferred through this disk, which is used as the primary transfer element. The cell measures and monitors differential heat flow. In developing a film for any type of coating, the effect of temperature is crucial, and the distinctive features of the performance of the coating are controlled by glass transition temperature. The transition is detected as a distinct increase in the rate of change of the polymers' thermoplasticity with temperature (Turi 1997).

The instrument was calibrated according to the manufacturer's procedure, using Indium as the standard reference material. Samples were weighed (approximately 8–10 mg) in aluminum pans, hermetically sealed, and loaded into the DSC cell. The glass transition temperature was determined using the midpoint temperature, which is the point on the thermal curve corresponding to one half of the heat flow difference between the extrapolated onset point of transition and the end inflection change (ASTM 1997a). Experiments were performed on the cured films at a heating rate of 10 °C/min from -25 °C to 200 °C in a nitrogen atmosphere. Samples from the 120-mm cartridge were lightly scraped with clean razor blades.

The samples from the coated plastic strips basically used for film thickness control in production were selected to establish a baseline. These samples were previously punched, and the clear plastic strip was used as reference. To duplicate results, DMA was performed on several strips, but the results were inconsistent because of material variation from the clear plastic strips. Although there are various ways to determine  $T_g$ , and other techniques were

considered, DSC seems to be the more practical one in this case. The coatings on the plastic strips were scraped with a razor, and experiments were performed on the cured films at a heating rate of 10 °C/min from -25 °C to 200 °C.

**3.3 FTIR.** An FTIR spectrometer (model 5DXB, Nicolet Instruments, Madison, WI) was used to collect and process infrared wavelength absorbance/transmission spectra of the specimens. The spectrometer radiates a broad band of infrared light through the specimen. Depending on their chemical bonding, individual materials will absorb, transmit, or reflect the infrared (IR) light of various wavelengths. From the spectrum produced, information about chemical bonding is obtained from the location of group frequency peaks (Haslam 1965). Most spectra contain additional “fingerprint” peaks that are unique to a particular molecular structure. This technique is appropriate for all qualitative and some quantitative analyses of most materials. An IR spectrum is capable of indicating the chemical composition and/or bonding of organic, polymeric, and many inorganic substances (Haslam 1965). In this case, the FTIR was used to detect the specific functional epoxy groups remaining in a cured coating film. The epoxy resin of interest is commonly encountered in the coatings industry and is found in many formulations. Epoxy resins contain one or more terminal epoxy groups in their molecules, which appear in the IR trace as a low-intensity band at 10.9  $\mu$  ( $915\text{ cm}^{-1}$ ) (Haslam 1965). Monitoring this absorbance band is useful in estimating the degree of cure. As the coating cures, the terminal epoxy ring opens, resulting in a reduction and/or loss of intensity at this frequency (Tatsumiya et al. 1997). During cure, the terminal epoxy group disappears, and their appearance/disappearance can be followed by their intensity. Furthermore, to aid in the identification process, the specimen’s spectrum is compared with a computer database of standard polymeric materials from an IR reference library.

**3.4 FTIR Application.** An attenuated total reflectance (ATR) accessory was attached to the FTIR for the initial analysis. This surface measuring technique involves placing the sample in direct contact with an internally reflecting crystalline plate. For this analysis, the crystalline plate was composed of zinc selenide (ZnSe). The samples were prepared for analysis by stripping the cured coating film from the casing and clamping it onto the crystalline plate.

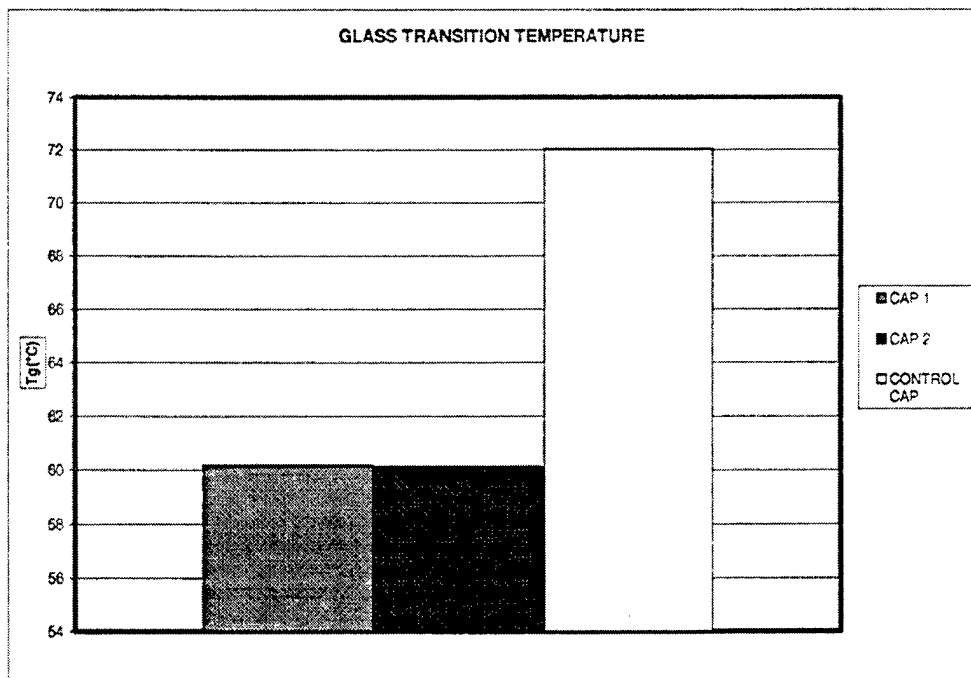
Sufficient pressure was applied to hold the sample in intimate contact with the crystalline plates for direct analysis. Additionally, an IR analysis was run on residual coated materials. The residues were thoroughly mixed with potassium bromide (KBr) and Tetrahydrofuran (THF), then oven dried at 105 °C to drive off residual solvents. The dried material was compressed into thin KBr pellets for spectral analysis. A blank KBr pellet was used as a background spectrum.

## **4. DSC Results**

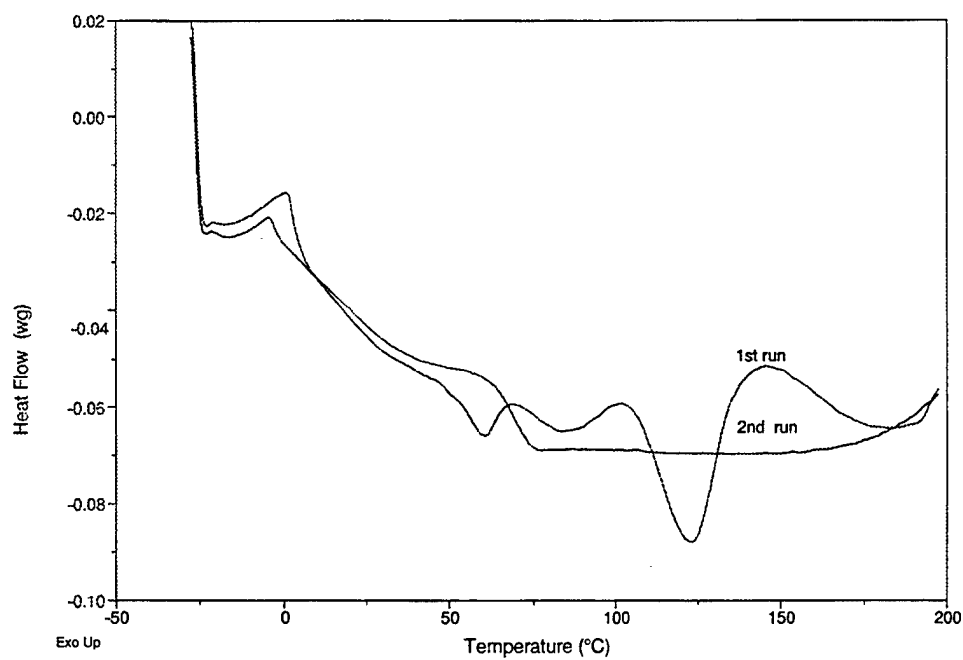
**4.1 Combustible Cartridge Cap Study.** A control cap was applied from an acceptable production lot and two caps from production lots that showed excess residue. These caps hold the projectile and are attached with adhesives to the cartridge casing. Figure 1 shows samples labeled as cap No. 1 and cap No. 2 with low Tg values, an average of 60.1 °C and 60.2 °C. The control cap as had an average of 72.0 °C. The significant differences in Tg between cap No. 1, cap No. 2, and the control indicates deviations in stoichiometry of component A to component B. Errors can occur either during the production of A and B or during the mixing process. Figure 2 shows typical DSC scans for these coatings. Experiments were performed on the cured film at a heating rate of 10 °C/min from -25 °C to 200 °C in a nitrogen atmosphere. Each sample had two runs. The first run erased thermal history and established the baseline; however, the recorded Tg was from the second run.

**4.2 Topcoat Study.** The difference in Tg values for the caps and control samples prepared in the laboratory are shown in Figure 3. A comparison of DSC scans for the residue, caps, and laboratory prepared samples are shown in Figure 4. The Tg values of 71–73 °C for the gun residue compares favorably with results for the control cap, indicating that this lot of paint was mixed to correct ratios. A lower Tg would indicate more polyamide and less epoxy, so the coating would be more flexible and less hard.

**4.3 Plastic Strips.** During the spray application, narrow strips of flexible plastic were coated with basecoat only, topcoat only, and a combination of both coats on dummy rounds to adjust the film thickness. The initial effort concentrated primarily on the topcoat because the



**Figure 1. Glass Transition Temperature of Control Cap and Caps 1 and 2.**



**Figure 2. Typical DSC Scans of Epoxy Topcoat.**

## Glass Transition Temperature

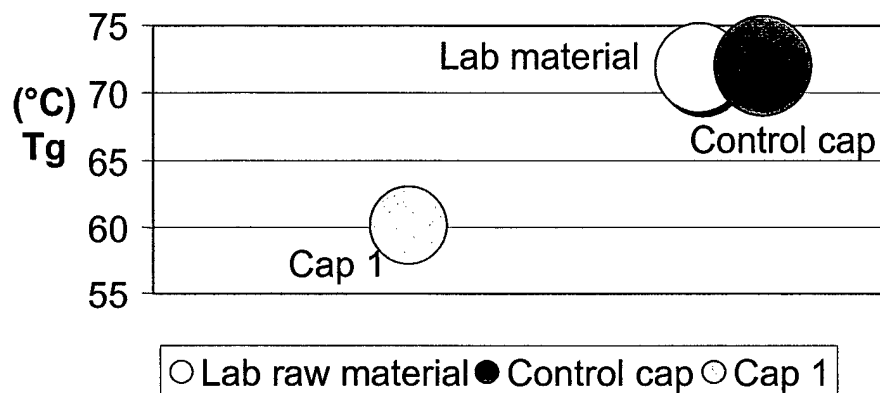


Figure 3. A Comparison of Caps With Laboratory Material.

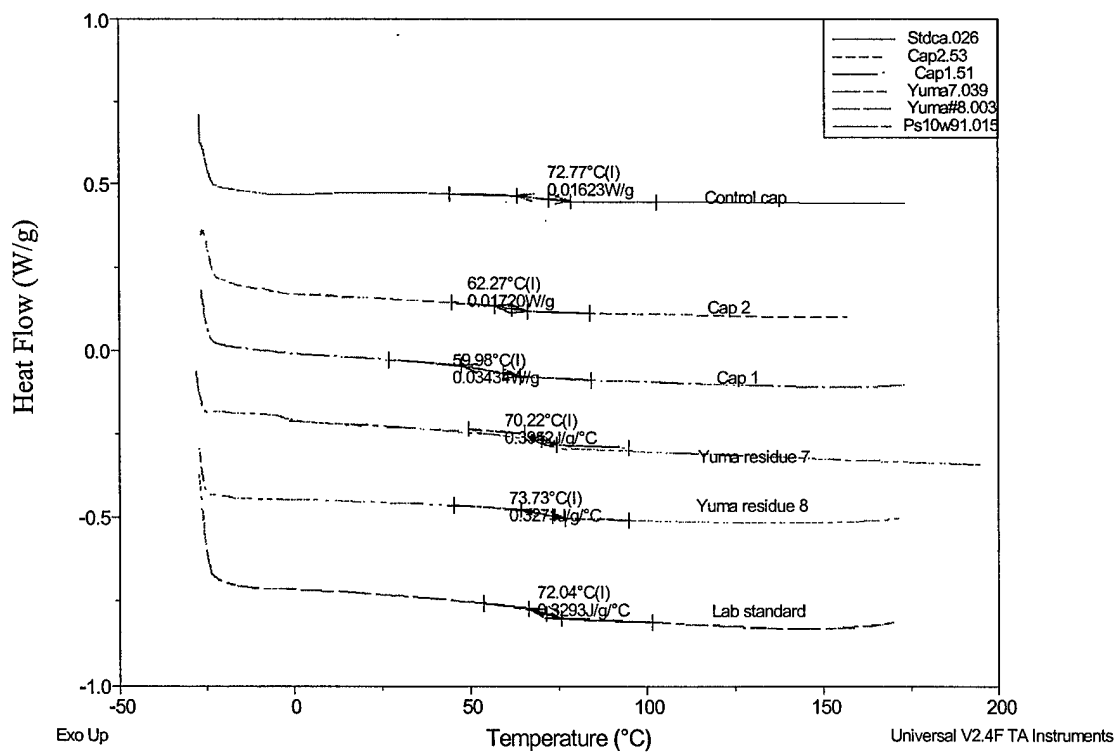
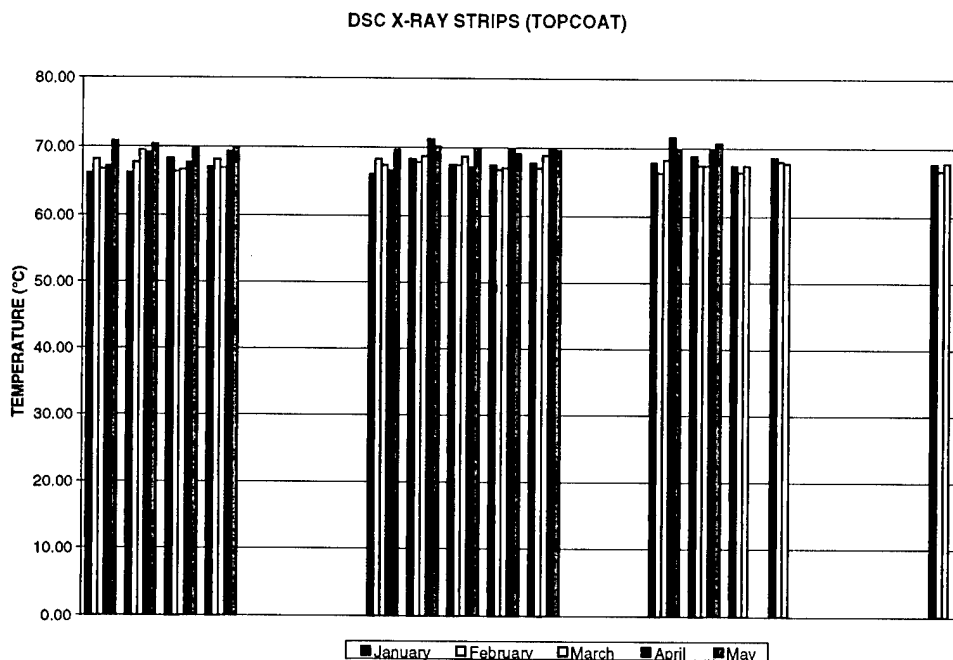


Figure 4. A Comparison of Caps, Residue, and Laboratory Control.

residue that remained in the chamber after firing was a relatively clear film with minor amounts of aluminum from the basecoat. These plastic production strips have been analyzed on a daily basis for several months to determine precision and methodology.

The average Tg value is shown in Figure 5. Each column in the graph represents a production batch. From January 1999–May 1999, approximately 70 plastic strips with Tg values ranging from 66 °C to 72 °C have been analyzed.



**Figure 5. Tg of Various Plastic Strips.**

#### **4.4 Retain Samples.**

**4.4.1 Room Temperature vs. Tg.** Retain samples prepared and air dried in the laboratory had Tg values in the upper sixties. The difference between sample A and sample B is the production lot of the polyamide. Since samples were tested over time from 4 days to 17 days, Tg increased over time and appeared to level off after approximately 17 days. Values for these

samples are shown in Table 3. This data indicate values that are within the defined parameter and also show that the composition of the retain sample is acceptable.

**Table 3. Room Temperature vs. Glass Transition Temperature**

Sample ID	Cure Time at Room Temperature (days)	Sample A Tg (°C)	Sample B Tg (°C)
Wet retain samples	4	66.6	65.3
	8	67.2	68.7
Sample A and B	12	68.0	68.7
	14	68.8	68.7
	17	70.8	68.9

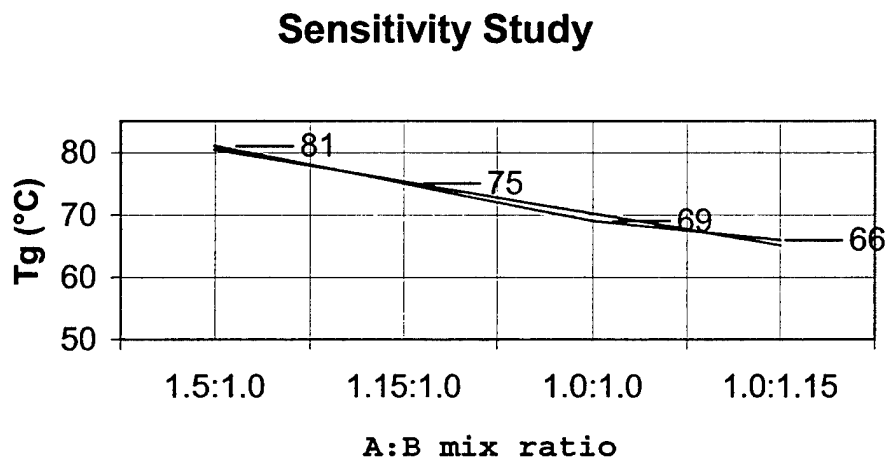
**4.4.2 Oven Cure Time vs. Tg (°C) and Pencil Hardness.** To determine the effect of time and temperature of force drying during production, samples were prepared in the laboratory and force dried at 160 °F for a length of time. Values for these samples are shown in Table 4. This data suggest that cure proceeds at 160 °F for a long time, and the final cure is proportional to high temperature and longer time. Although DSC results yield reliable Tg values at 160 °F for 30 min, these coatings were not fully cured. We also evaluated the coating using the American Society for Testing and Materials (ASTM) pencil test method for film hardness (ASTM 1999), which also verified inadequate cure. Since the ultimate properties of the epoxy network are achieved during the final stages of cure, it is critical that cure attain full completion. The pencil hardness scale is as follows:

Soft → → → Hard  
6B 5B 4B 2B B HB F H 2H 3H 4H 5H 6H

**Table 4. Tg (°C) and Pencil Hardness vs. Oven Cure Time**

Cure Temp. (min)	Pencil Hardness	Tg (°C)
160 °F (20)	HB	63.2
160 °F (30)	F	75.4
160 °F (40)	H	78.0

**4.4.3 Affected Cure Stoichiometry on Tg.** Since a shorter cure time leads to more residue, it was assumed that harder films and higher Tg would have less residue. To prove this, the ratio of epoxy to polyamide was varied. The expected glass transition temperature of the topcoat with component A/component B, at mix ratio of 1:1, should have a Tg of approximately  $70 \pm 2$  °C. A mix sensitivity study proved that even a 15% variation had adverse effects on Tg. Figure 6 displays the study.



**Figure 6. Tg of Epoxy Topcoat as a Function of A/B Mix Ratio.**

The primary vendors, Primex and Alliant Techsystems (ATK), also participated in analyzing these films. A correlation study was done on samples that were sprayed on aluminum panels at Alliant and air dried for 16 days. Good correlation was established between both laboratories with Tg results within 4 °C.



## 5. FTIR Results

Using the control cap as standard, the suspect cap No. 1 and residue from the firings were analyzed and compared. The resulting IR spectrum of each sample is shown in Figures 7 and 8. The ATR analysis of the control cap and the sample cap No. 1 indicate similar functional group composition with minor spectral differences. Also, as shown, the epoxy absorbance band at  $915\text{cm}^{-1}$  has a very low intensity, suggesting that the cure is relatively complete (Federation of Societies for Paint Technology 1969).

The residue from the firings was analyzed to identify its components. Figure 9 represents a typical IR spectrum of the residue. Analyzing the composition of the residue from the Yuma firing indicates that it has compositional characteristics similar to those of the control sample cap. When comparing spectra, the relative intensities of individual absorption and the overall intensities of the spectra could vary due to specimen thickness variation.

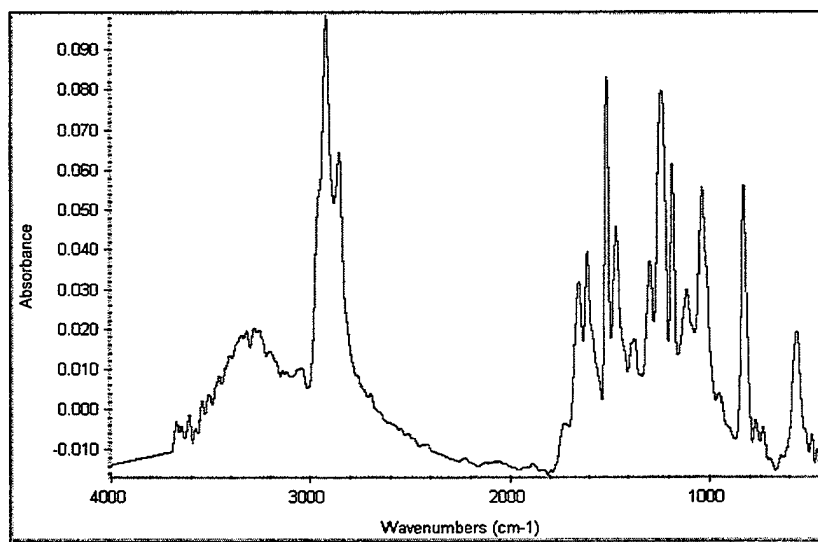
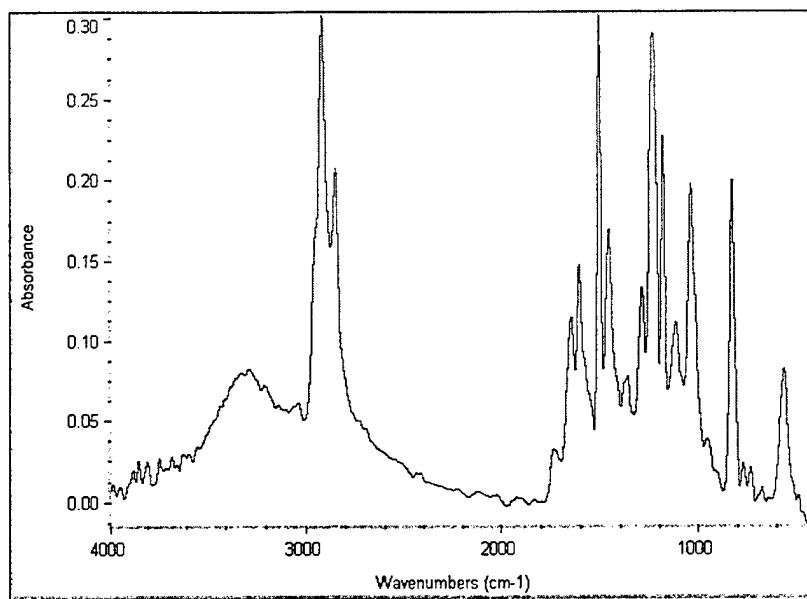
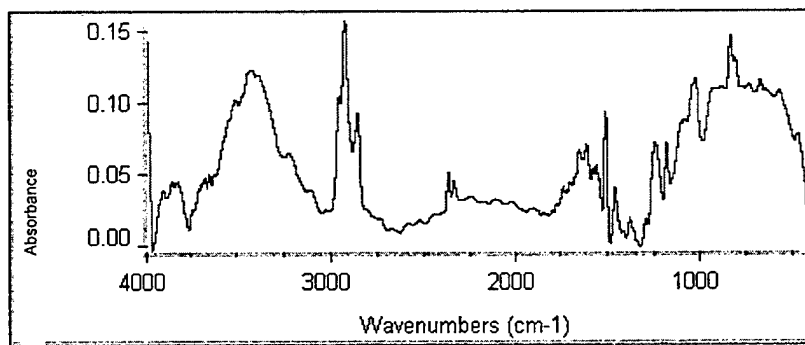


Figure 7. ATR Spectrum of Sample Cap.

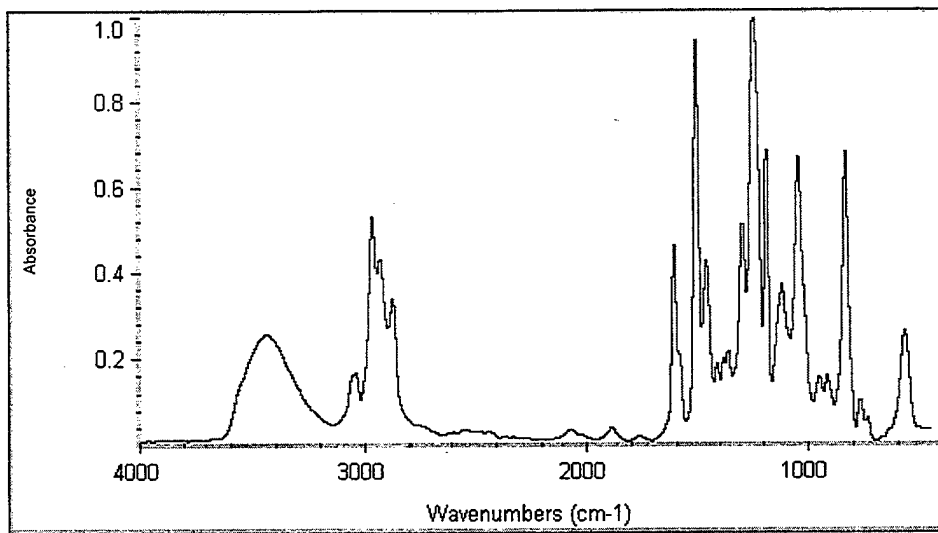


**Figure 8. ATR Spectrum of Control Cap.**



**Figure 9. FTIR Spectrum of Yuma Residue.**

Figure 10 is a reference spectrum of an uncured Bis-phenol A-Epoxy resin. A library search was done after each analysis, and this spectrum was a computer match to the samples in the library search.



**Figure 10. FTIR Spectrum of Bis-phenol A-Epoxy.**

## **6. Koenig Pendulum Hardness**

The wet retains and the laboratory controls were analyzed further to determine if a physical or mechanical preliminary screening test could indicate stoichiometric variations. Koenig testing was done to distinguish the difference between dry film thickness (DFT) and the various ranges of coating stoichiometry. The objective was to use the Koenig testing as a simple and quick screening prior to DSC testing; then, if these distinctions were possible, Koenig testing would complement the DSC technique.

The method for Koenig pendulum film hardness testing used by the ARL Coating Technology Team is the ASTM method (ASTM 1995). The procedure is based on a pendulum resting on a coated surface set into an oscillation motion. The Koenig hardness consists of both the damping time and the corresponding number of oscillations required for the amplitude to decrease from 6° to 3°.

The principle behind the test is based on the fact that the amplitude of oscillations decreases more rapidly when supported on softer films rather than on harder surfaces. A photo sensor is

accurately positioned to register swings of the pendulum. An automatic electronic counter registers the signals from the photo sensor, and the test result is shown on the digital display.

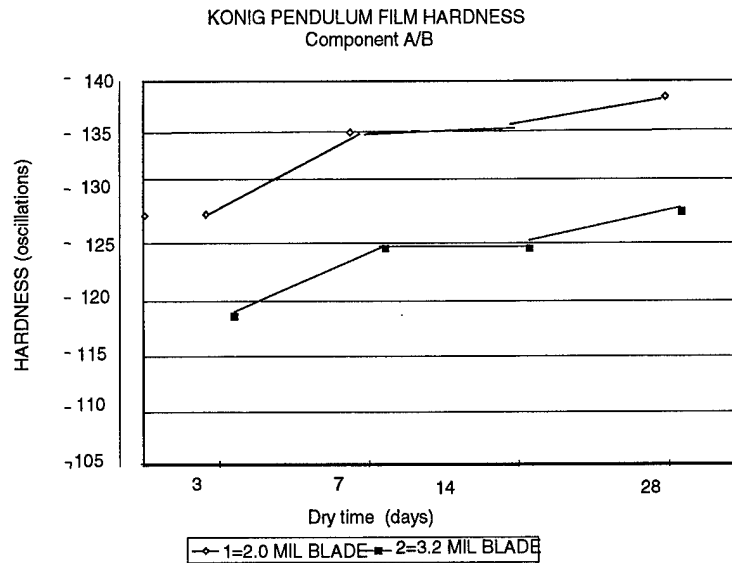
But this procedure is limited to certain conditions. The surface of the coating should be level and free of disturbances. Dust, poor leveling properties, and coarse pigment agglomerations can cause false measurements. The substrate should not be deformed or vibrate under the load of the pendulum. DFT is crucial and should be at least 1.2 mil (30.5  $\mu\text{m}$ ) to minimize substrate influences. Temperature and relative humidity must be carefully controlled.

The epoxy topcoat was applied at two different film thicknesses on glass panels and air dried at room temperature and humidity. The instrument used was a Byk-Gardner from Paul N. Gardner Co., Inc., model no. 5854. The Koenig apparatus was calibrated daily according to specifications. Hardness measurements were considered on cured sample films from 3 to 30 days.

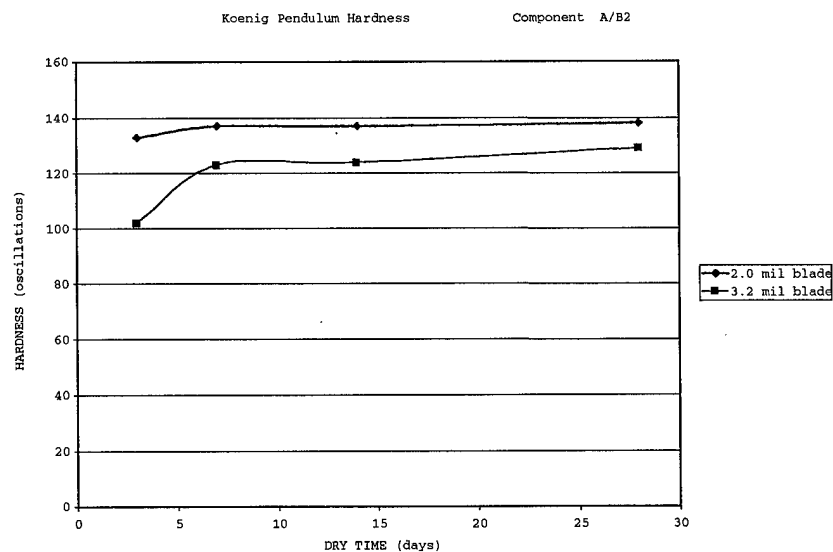
The graph in Figure 11 shows a typical pattern of the cure rate for both formulas; the laboratory formula is labeled component A/B. The thicker draw downs made using a 3.2-mil blade demonstrate a much slower cure rate than the draw downs using a 2.0-mil blade.

Figure 12 represents the cure rate of the component A/B2 formula. The difference between the two formulas is the production lot of the polyamide. The illustration confirms the cure rate is the same as that in Figure 11.

The laboratory control designated KC-86-1 was made using 100 parts by weight of epoxy to 45.0 parts by weight of polyamide; KC-87-1 was made using 100 parts by weight of epoxy to 52.5 parts by weight of polyamide.



**Figure 11. Component A/B.**



**Figure 12. Component A/B2.**

During the period (3–30 days), we could not tell the difference between 45.0 lb per hundred of resin (phr) and 52.5 phr was indistinguishable. This is clearly shown in Figure 13, where the two lines are indistinguishable. Therefore, the Koenig test, though simple and quick, was not a good screening test for the DSC test.

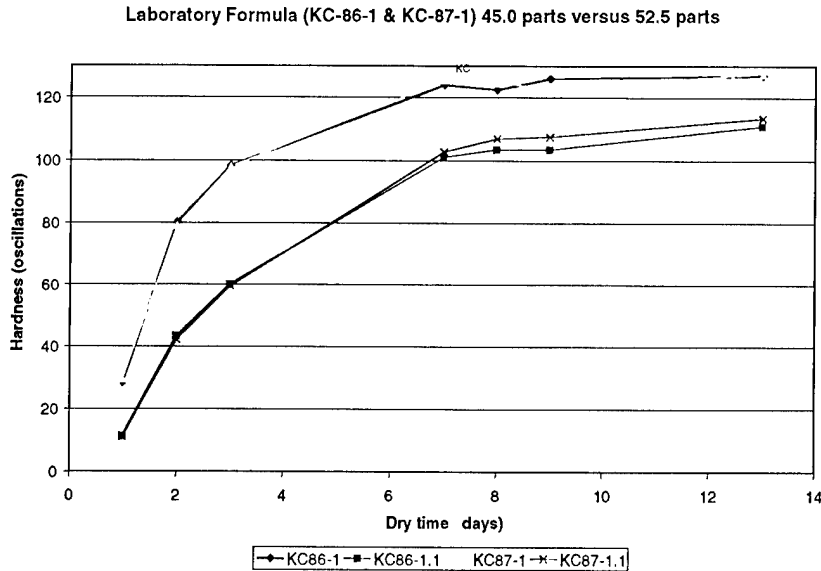


Figure 13. Formula Comparison of 52 phr to 45 phr.

## 7. Conclusions

The current epoxy coating was developed to offer maximum film deformation resistance from normal usage, to ensure resistance to moisture, and to minimize any residues left in the gun chamber from previous firings. The ARL-developed coating has met all of these requirements. However, sufficient data does not exist to compare variables in coating formulations to the amount of residue left after firings. It has been noted that residue is more prevalent when “hot” (120 °F) rounds are fired and when the rounds are fired only seven days after applying the coating. A few weeks later, these same rounds show minimal residue.

To the formulator, this indicates that the current system has sufficient toughness and flexibility; however, with some modification to a higher T<sub>g</sub> value, residue would be minimized when the rounds are fired at hot temperatures. This could be accomplished by adjusting the topcoat resin ratios from 52.5 to 45 parts by weight of polyamide per 100 parts of the epoxy resin.

DSC is a valuable quality control tool for tracking cure vs. time, provided that the ratios of epoxy and polyamides are consistent. This thermal study showed that incomplete film curing and higher temperature are the primary causes of gun tube residue. Based on the DSC data and observations at field testing, rounds should be allowed to cure for a minimum of three weeks before firing.

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE September 2000		3. REPORT TYPE AND DATES COVERED Final, Nov 98 - Jun 99
4. TITLE AND SUBTITLE Investigation of Residue and Coating Stoichiometry on 120-mm Combustible Cartridge Cases			5. FUNDING NUMBERS  622624H1800	
6. AUTHOR(S)  Kestutis G. Chesonis, Pauline M. Smith, and William S. Lum				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Research Laboratory ATTN: AMSRL-WM-MA Aberdeen Proving Ground, MD 21005-5066			8. PERFORMING ORGANIZATION REPORT NUMBER  ARL-TR-2337	
9. SPONSORING/MONITORING AGENCY NAMES(S) AND ADDRESS(ES)			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) <p>An investigation was conducted to determine the cause of coating residue found in the test gun chambers during qualification firing of 120-mm combustible cartridge case (CCC) ammunition for the M1A1/A2 main battle tank. The CCC is coated with a clear epoxy-polyamide topcoat and a highly pigmented aluminum epoxy basecoat. A laboratory analysis was performed on actual residues; parts of the CCC retain samples of actual coatings and laboratory control samples. Since there are six different resins used in this two-component coating system, production errors and deviations from correct mixing prior to application could have caused the residue. Any variation from the specified stoichiometry could also result in films with lower cross-link densities and lower glass transition temperatures (T<sub>g</sub>). Rounds fired at 120 °C have more residues than identical rounds fired at ambient temperature; additional weeks of cure lower the amounts of residue. A differential screening calorimeter (DSC) analysis of the various samples revealed that a suspect lot of ammunition with higher levels of residue had lower T<sub>g</sub> values. Fourier-transform infrared (FTIR) was effective in monitoring the degree of cure of the epoxy coating system. Analyzing the residue from actual firings showed similar features. Koenig hardness values showed variations caused by dry film thickness, but also showed insignificant changes caused by transformations in the stoichiometry of the clear topcoat from 52.5 to 45 phr (pounds per hundred of resin).</p>				
14. SUBJECT TERMS 120-mm combustible cartridge case, epoxy coating, DSC, T <sub>g</sub> , Koenig Hardness Tester, FTIR, ammunition			15. NUMBER OF PAGES 27	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	

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